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# PHYSICAL PARAMETERS IN SYNTHOIL PROCESS

Quarterly Report for the Period  
October–December 1975

by

J. Fischer, R. Lo, S. Nandi,  
D. Fredrickson, T. Bump,  
T. Mulcahey, H. Huang, and A. Jonke

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J. Fischer, R. Lo, S. Nandi,\*  
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T. Mulcahey,\*\* H. Huang,  
and A. Jonke

Chemical Engineering Division

Previous report

ANL-75-76      July—September 1975

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## PHYSICAL PARAMETERS IN SYNTHOIL PROCESS

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J. Fischer, R. Lo, S. Nandi, D. Fredrickson,  
T. Bump, T. Mulcahey, H. Huang, and A. Jonke

## ABSTRACT

This work is being done in support of the development of processes for converting coal to liquid fuel of low sulfur content, suitable for use in power production. Most of the effort is intended to produce information applicable to the SYNTHOIL process.

In the SYNTHOIL process for converting coal to a low-sulfur fuel oil, coal is liquefied and hydrodesulfurized in a turbulent-flow, catalytic packed-bed reactor. First, a slurry of coal in recycled oil is reacted with hydrogen at 450°C and 2,000-4,000 psi in the presence of Co-Mo/SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> catalyst. The turbulent flow of fluid prevents the coal's mineral matter from settling and plugging the reactor. Then, the gross liquid products are centrifuged to remove the unreacted solids. The centrifuged liquid product is a low-sulfur, low-ash fuel.

The work for this program includes four tasks.

Task I, Heat of Reaction of Hydrogen with Coal Slurries.

Determination of the heat of reaction of coal slurries with hydrogen, with and without catalysts, at 2,000-4,000 psi and 400-475°C. Specifications for a calorimeter have been completed.

Task II, Heat Transfer Coefficient. Determination of the coefficients of heat transfer from SYNTHOIL reactor fluids to heat exchangers in the following ranges of conditions: pressure, 2000-4000 psi; temperature, to 475°C. Design of the test unit is 75% completed.

Task III, Additives to Facilitate Separation of Solids from Liquids. Investigation of additives to facilitate the removal of solids from oil produced in coal liquefaction processes. Additives being considered are those which would act as agglomerating or flocculating agents and/or also would alter the physical properties of the mixture, improving separation in subsequent steps. The viscosity of SYNTHOIL product oil has been measured between 50 and 200°C. The viscosity of the oil changed when it was exposed to air. The oil also exhibited non-Newtonian viscosity behavior.

Task IV, Catalyst Testing. Testing of new catalysts in 300-hr runs in a continuous high-pressure coal liquefaction unit to operate at 2000-4000 psi and 450°C at a minimum feed rate of 4 oz/hr of coal slurry containing 35-50% coal. The product samples will be collected and analyzed for: coal conversion; sulfur, nitrogen, and oxygen removal; hydrogen consumption; viscosity; and specific gravity of the product oil. Design of the test unit has been completed.



## SUMMARY

Heat of Reaction of Hydrogen with Coal Slurries  
(S. Nandi and D. Fredrickson)

The objective of this task is to obtain heat-of-reaction data for the hydrogenation of coal slurries typical of those used in the SYNTHOIL process under SYNTHOIL process conditions. The heat release data is required for proper design of the slurry preheater and the reactor of the SYNTHOIL plant.

To accomplish this task, the reaction will be carried out in a calorimetric pressure vessel at 2000-4000 psi up to 475°C. Specifications for the apparatus have been completed and a contract for its fabrication is being prepared.

Heat Transfer Coefficient  
(T. P. Mulcahey and T. R. Bump)

Work on this program was initiated in September 1975 to provide measured coefficients of heat transfer from SYNTHOIL process feed and effluent products to container walls, for use in the future design of heat transfer equipment for the process. Preliminary unit configuration and component sizing of a test unit were completed, based on the slurry and hydrogen flow rates being comparable to those in a 1/2 ton/day plant, *i.e.*, 25 lb/hr of slurry feed and 1300-scfh hydrogen flow. After initial estimates indicated that the cost incurred for a 1/2 ton/day unit would be larger than anticipated previously, the feasibility of reducing construction costs by combining the heat transfer test unit and the catalyst test unit (Section IV of this report) into a single multiple-purpose unit was investigated. The results of the investigation indicated that a multiple-purpose unit would be technically feasible; however, if it were constructed, there would be no time in the schedule for repeating heat transfer or catalyst test runs or for any unexpected repair, and the experimental flexibility would be limited. It was concluded that it will be best to construct two separate units, as originally planned.

In order to expedite purchase and construction, long-lead procurement items have been ordered and work has progressed on unit design.

Additives to Facilitate Separation of Solids from Liquids  
(H. Huang)

The objective of this task is to investigate the effect of additives in facilitating the removal of solids from oil produced in coal liquefaction processes. Additives being considered are those which would act as agglomerating or flocculating agents and/or would alter the physical properties of the mixture, improving separation in subsequent steps.

During this period, we have received from the Pittsburgh Energy Research Center (PERC) two one-gallon samples of SYNTHOIL centrifuged product oil and two one-gallon samples of SYNTHOIL uncentrifuged product for this study. The viscosity of the centrifuged oil has been measured between 50 and 200°C, using a Brookfield Model LVT viscometer along with a Brookfield Thermosel System. We observed changes in viscosity of the oil upon exposure to air, as well as non-Newtonian viscosity behavior.

Determinations of particle-size distribution and ash content of SYNTHOIL product oils are planned.

### Catalyst Testing

The objective of this task is to evaluate new commercially available catalysts for use in the SYNTHOIL process. A continuous high-pressure coal liquefaction unit will be constructed to operate at SYNTHOIL process conditions --450°C and 2000 to 4000 psi. Catalysts will be tested in 300-hr continuous runs. The evaluation will be made on the basis of analyses of coal input and product samples. Samples collected at least once every 24 hr will be analyzed to obtain information on: percent coal conversion; sulfur, nitrogen, and oxygen removal; hydrogen consumption; oil viscosity; and specific gravity of the product oil.

Design of the catalyst test unit has been completed, and components which have long delivery times have been ordered. A purchase requisition for the unit has been issued. It is estimated that installation of the unit will be completed by the end of August, 1976.

# I. HEAT OF REACTION OF HYDROGEN WITH COAL SLURRIES (S. Nandi and D. Fredrickson)

The objective of this program is to obtain heat of reaction data for the hydrogenation of coal slurries typical of those used in the SYNTHOIL process, under SYNTHOIL process conditions. The heat release data is required for proper design of the slurry preheater and the reactor of the SYNTHOIL plant.

In the preceding quarterly report (ANL-75-76), several general approaches were reviewed that might be suitable for this work. We have concluded that to accomplish this task, the reaction should be conducted in a pressure vessel, at 2000 to 4000 psi and up to 475°C. The pressure vessel should be contained in a calorimeter for measuring the heat evolved.

We have therefore developed a set of specifications for the design and ultimate building of the calorimeter. The specifications are summarized below:

- a. The system shall be capable of measuring the heat evolved due to the reaction of hydrogen with a coal-oil slurry within the ranges, 2000 to 4000 psi and 375 to 475°C.
- b. The system shall have a provision for introducing coal slurry samples (held at ambient temperature) via a drop tube into the pressure vessel while it is operated at the maximum temperature and pressure.
- c. The maximum operating temperature of the system shall be 500°C.
- d. The maximum pressure shall be 4000 psi.
- e. The system shall be capable of measuring the heat evolved from 20 to 50 g samples.

The contract for the design and building of the calorimeter has not yet been awarded. We can, however, give a general idea (subject to change) of what the apparatus will consist of and the operating procedure.

The method selected is to measure the heat evolved while the reaction (hydrogenation) is conducted in a constant-volume system. The extent of reaction (hydrogen consumption) is then obtained from the hydrogen balance between the reactants and products.

It is proposed that the apparatus consist of a one-liter pressure vessel to which a drop tube is attached. The pressure vessel will be surrounded by a calorimeter consisting of an aluminum block, to which heaters and temperature sensors will be attached.

The aluminum block, in turn, will be inside an enclosure for proper thermal insulation. The power supply and temperature control of the aluminum block will be so designed that any heat evolved or absorbed in the pressure vessel will cause a change in the power consumption which will be accurately measured by monitoring the current and voltage.



The proposed method of operation is to place a weighed amount of coal slurry (with or without catalyst), contained in a polyethylene bag, in the cold portion of the drop tube. The sample will be so located that a heat shield will protect it from the heated zone with the sample in place. The system will be evacuated, after which hydrogen will be introduced into the pressure vessel to the desired pressure (at ambient temperature). The amount of hydrogen that has been charged will be calculated from the known volume and pressure of the system. The pressure vessel will then be brought to the desired temperature. When a steady state is achieved, the coal slurry will be dropped into the pressure vessel and stirring will be started. Monitoring of the power input from this time (sample addition) to the time when steady state is again obtained will give some of the necessary information from which the heat of reaction will be calculated. After the reaction is complete, the system will be allowed to cool to about 110°C. The gas inside the apparatus will be passed through a number of weighed traps and will be metered; a portion will be collected for analysis. After this step, the system will be allowed to cool to room temperature and the remainder of the products will be collected. The elemental assays of the collected gas, liquid, and solid products will be determined. From the known analysis of the starting coal slurry, the hydrogen consumed in the reaction will be calculated.

At present, ANL personnel are working out the details for awarding the contract to the company we feel is best qualified to construct the apparatus. It is estimated that nine months will be needed for fabrication of the system once the contract has been agreed to, which means that installation would start in November, 1976.

## II. HEAT TRANSFER COEFFICIENT (T. Mulcahey and T. Bump)

The objectives of this task are to determine heat transfer coefficients applicable to the SYNTHOIL process feed heat exchangers and effluent heat exchangers and to identify mechanisms for the heat transfer process. Information will be supplied, in a form suitable for SYNTHOIL heat exchanger design, on heat transfer film coefficients for (a) SYNTHOIL reactor effluent cooled by a metal surface and (b) SYNTHOIL feed heated by a metal surface. The following ranges of conditions will be covered:

Pressure:	2,000-4,000 psig
Temperature:	200-475°C
Flow of Fluids:	Turbulent

The experimental work will be carried out in a 1/2-ton-per-day heat transfer test loop which will include a slurry tank, feed pump, heater, cooler, letdown tanks, and power, flowrate, and temperature instrumentation. Three different coals suitable for SYNTHOIL processing will be studied in the investigation. Theoretical modeling of the heat transfer process will be attempted, correlating primary interaction of the liquid, particles, and walls. The resulting model should be capable of describing the heat transfer rates, as well as the temperature history, of the particles in the slurry.

Work was initiated in September 1975 on this program to provide measured heat transfer coefficients of the SYNTHOIL process feed and effluent products for use in future design of heat transfer equipment for the process. Preliminary unit configuration and component sizing of a test unit was completed

based on the slurry and hydrogen flowrates being comparable to those in a 1/2-ton/day plant, *i.e.*, 25 lb/hr of slurry feed and 1300 scfh hydrogen flow.

Since initial estimates indicated that a larger cost than anticipated would be incurred for a 1/2 ton/day unit, reducing construction costs by combining the heat transfer unit and catalyst unit (Section IV) into a single multiple-purpose unit was investigated. The results of the investigation indicated that it would be technically feasible; however, if it were constructed, there would be no time in the schedule for repeating heat transfer or catalyst test runs or for any unexpected repairs, and the experimental flexibility would be limited. It was concluded that it will be best to construct two separate units as originally planned.

Procurement of Long-Lead Items. In order to expedite the purchase of components and to prevent delays in construction, long-lead procurement items were ordered while work was progressing on unit design. The major long-lead items currently on order are described below.

1. Low-pressure slurry circulation and feed pump. This is a progressing cavity (screw) type pump of stainless steel construction with a capacity of 50 gph at pressures ranging from 50 to 100 psi. This pump supplies agitation and mixing to ensure slurry suspension; it also supplies a high suction pressure to the high-pressure slurry pump to prevent loss of pump prime. This pump accepts from and discharges to the 10-gal slurry feed tank.

2. High-pressure slurry feed pump. This is a piston, positive-displacement pump which accepts up to 5 gph of slurry from the low-pressure slurry feed pump loop and injects it at high pressure (up to 4400 psi) into a hydrogen stream to form a gas-slurry mixture for subsequent heating and cooling in the heat transfer test section. This is a stainless steel pump with a ceramic plunger, and it is rated for 95°C service.

3. High-pressure wash-water pump. This is identical to the high-pressure slurry pump and is used to inject water into the effluent gas stream to assist in removing the ammonia and ammonium salts that may be deposited while the gas stream is cooled from 100°C to slightly above room temperature.

4. High-pressure hydrogen-transfer compressor. This is a compressor which recirculates hydrogen (also nitrogen and helium) around the loop through the test section. The rate of transfer varies as the suction pressure to the compressor is varied; at a suction pressure of 2800 psi the compressor will circulate up to 1500 scfh through the loop at 4500-psi pressure.

5. High-pressure hydrogen-makeup compressor. This compressor is used to increase the pressure of hydrogen (from tanks at pressures of 500 psi and below) to the high pressure of the system. Makeup gas is needed to replace hydrogen absorbed by the coal and oil and to replace liquid volume during high-pressure blowdown of the effluent products. The compressor ordered is similar to the hydrogen-transfer compressor (item 4) and supplies 140 scfh of makeup gas.

6. High-pressure receiver. This vessel is used to collect the 150°C liquid effluent product as it exits from the test section. It contains baffle plates to separate gas-entrained liquid and has a 5-gal capacity.

Periodically, the collected liquid is removed from the high-pressure receiver to the low-pressure receiver. The tank is a vessel with a diameter of less than 6 in., designed for use at 5000 psi and 315°C.

7. High-pressure water-wash receiver. This vessel collects the wash water and salts obtained by scrubbing the effluent gas coming from the high-pressure effluent receiver. The tank is similar to the high-pressure effluent receiver.

Description of Heat Transfer Units. The heat transfer coefficient test unit will be a modified higher-capacity version of the catalyst test unit described in Part IV of this report. In the transfer coefficient test unit, heating and cooling test sections will be provided for measurement of heat transfer coefficients instead of preheat and catalyst test sections.

There will only be one slurry pump. A slurry makeup system will be added for blending of relatively large amounts of coal tar-derived products and coal to form the coal slurry feed material for the heat transfer unit.

Test Section Description. The test section will consist of a heating section and a cooling section. In the heating section, tubing containing the slurry-gas mixture will be heated by direct electrical resistance heating. Thermocouples will be attached to the outside wall of the tubing. The calculated temperature at the inside wall of the tubing will serve as the basis for determining the film coefficient of the slurry-gas mixture. At steady state, the heat transferred from the tubing to the slurry will be the electrical heat input (except for small losses). The temperature of the slurry-gas mixture will be measured at the inlet and outlet of individual heating elements. The number of heating elements used will depend on the cost of design and fabrication; current plans are to use four physically identical heating elements. The heat flux will be varied in each section to maintain the wall temperature below 465°C. It is known that charring of the slurry starts at temperatures above 465°C.

The conceptual design of the cooling portion of the heat transfer test section has not yet been fixed. The feasibility of using air as the coolant medium is being investigated because of numerous advantages of its use. Indications are that air cooling using four cooling elements is feasible. The high-temperature part of the cooling system may consist of straight tubing with the three lower-temperature sections consisting of coiled tubing. The outlet temperature of the test section effluent will be 150°C. The subdivision of the heat transfer test section into a section with four heating elements and a section with four cooling elements will allow the average heat transfer coefficients to be obtained over specific temperature ranges.

The specifications for the procurement and assembly of all components of the heat transfer coefficient test unit except the test section have been written and are under review. The detailed specifications for the test section are being developed; completion of these specifications in January 1976 is expected. Some available high-current low-voltage power supplies which may be used for heating the test section have been located, and transfer of this equipment is being requested.



Oil for Slurry Makeup. Operational problems mentioned in the preceding quarterly report indicated that not enough SYNTHOIL effluent product is available to allow operation of the heat transfer measurement unit with a SYNTHOIL slurry. Therefore, coal tar distillates will be blended to produce a material similar to SYNTHOIL. Then, a coal slurry will be made from the blended oil.

PERC Review. The proposed designs of the heat transfer coefficient test unit and the catalyst test unit were reviewed at the Pittsburgh Energy Research Center, whereupon the following changes (some of the components are described above) were made to the heat transfer unit:

1. The volume of the high-pressure receiver was reduced from 15 gal to 5 gal (the same size as is used in the catalyst test unit), and a device was added for automatic blowdown of liquid product from the high-pressure receiver.
2. A device was added to wash the gas leaving the high-pressure receiver.
3. A change was made in the heating test section to limit the maximum film temperature to the 460°-465°C range.
4. Hydrogen makeup rate was increased to allow a more flexible test program to be run, *i.e.*, by allowing both steady state data and slowly varying dynamic data to be taken.
5. Internal baffles were provided in the high-pressure receiver to help prevent entrained liquid from being carried out of the high-pressure receiver.
6. Hand-operated high-pressure pumps for unplugging the system were included.
7. A slurry makeup system was added in which coal tar products will be blended to form a liquid substitute for SYNTHOIL in coal slurry feed material. The slurry makeup system will also function as a facility where (a) the oil and coal will be thoroughly mixed to wet the coal with the oil and (b) a large quantity of slurry can be stored, permitting batches to be made once per day or less often and thus providing feed material having more uniform properties.

### III. ADDITIVES FOR SEPARATION OF SOLIDS FROM LIQUIDS (H. Huang)

The objective of this task is to investigate the use of additives to facilitate the removal of solids from oil produced in coal liquefaction processes. These additives should be able to act as agglomerating or flocculating agents and/or to alter the physical properties of the mixture so as to improve separation in subsequent steps. The properties which may be altered and for which experimental data will be collected are viscosity, interfacial tension, and flocculation characteristics.

Information was given in our preceding quarterly report (ANL-75-76) on separation techniques under development at the Pittsburgh Energy Research Center (PERC) and on planned ANL work on physical property modification and flocculation. In this report, specific information about the experiments is presented.

## Viscosity and Flow-Property Measurement of Coal-Liquefaction Product

We received two one-gallon samples of SYNTHOIL centrifuged oil and two one-gallon samples of SYNTHOIL uncentrifuged oil for this study from PERC on December 1, 1975. This product was made from West Virginia coal and had been stored in press-cap metal cans.

Since the viscosity of a slurry significantly affects performance in solid-liquid separation, the viscosity before and after chemical pretreatment needs to be known accurately. A Brookfield Model LVT Synchro-electric viscometer is being used to measure the viscosity of the coal-liquefaction oil. This viscometer, along with a Brookfield Thermosel System, permits viscosities of liquid samples between 5 and 10,000 cp to be measured with a precision of 1%, at temperatures between ambient and 260°C. Due to the many speeds at which the spindles of this viscometer can rotate, flow properties of the liquid samples are being determined quickly and easily. The temperature inside the liquid-sample container of the Thermosel has been measured with a calibrated copper-constantan thermocouple and has agreed with the set temperature (degrees, Centigrade) on the controller within 1%.

Viscosity of centrifuged SYNTHOIL oil has been measured over a temperature range of 50 to 200°C (Fig. 1). The viscosity of this oil (15 cp at 150°C and 2000 cp at 60°C) is lower than that for the unfiltered oil from the COED coal-liquefaction process (20 cp at 150°C and 50,000 cp at 60°C).<sup>1</sup> Also shown in Fig. 1 is the viscosity of the same oil after it had been exposed to air for 60 hr. The latter measurements give a value of 160 cp compared with 86 cp for the sample not exposed to air at 100°C, and the corresponding values at 200°C are 7.5 cp and 6.5 cp. These differences are possibly due to oxidation of the sample.

During viscosity measurement, the flow behavior of the centrifuged SYNTHOIL oil was also investigated by varying the rotational speeds of the viscometer spindles at constant temperature. Figure 2 gives two representative plots of the viscosity variations at various shear rates. For either isotherm, the data at the lower shear rate differ from those at the higher shear rate by as much as 30%. This non-Newtonian behavior had not been observed for COED unfiltered oil.<sup>1</sup> Of the measurements used to produce Fig. 1, all except one were taken at a shear rate of 40 sec<sup>-1</sup>; the data point at 50°C was measured at a shear rate of 0.4 sec<sup>-1</sup>.

## Flocculation Test

Two methods that may be suitable for the evaluation of flocculants are determination of the settling rate and the size of the flocs. When graduated cylinders were used as settling columns for SYNTHOIL oil, the level of the particle-fluid interface could not be accurately determined. The use of a settling column of small diameter might overcome this difficulty; however, in such a column the wall effect on the settling of particles would be significant and hard to assess. We therefore will investigate the transmittance of a point light source through thin layers of the SYNTHOIL oil to determine the position of the fluid-solid interface.

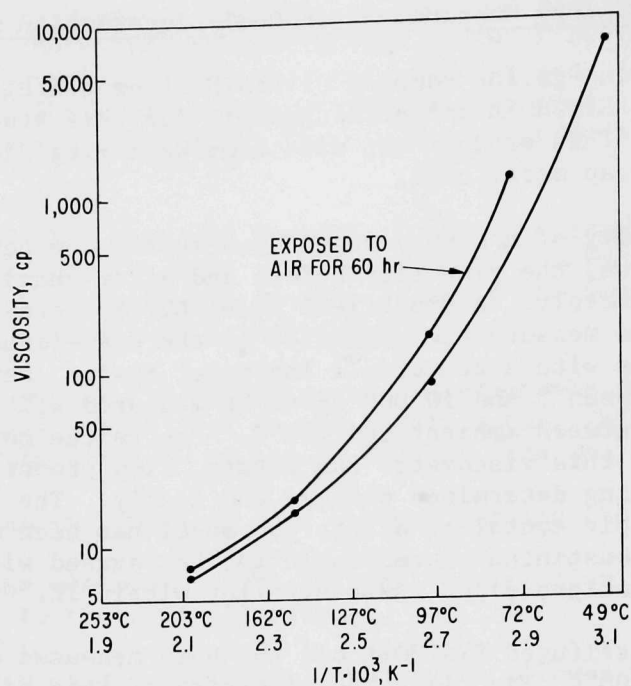


Fig. 1. Viscosity-Temperature Behavior of SYNTHOIL Centrifuged Oil. Shear Rate of  $40 \text{ sec}^{-1}$  Except for Point at  $50^\circ\text{C}$ , For Which Shear Rate Was  $0.4 \text{ sec}^{-1}$

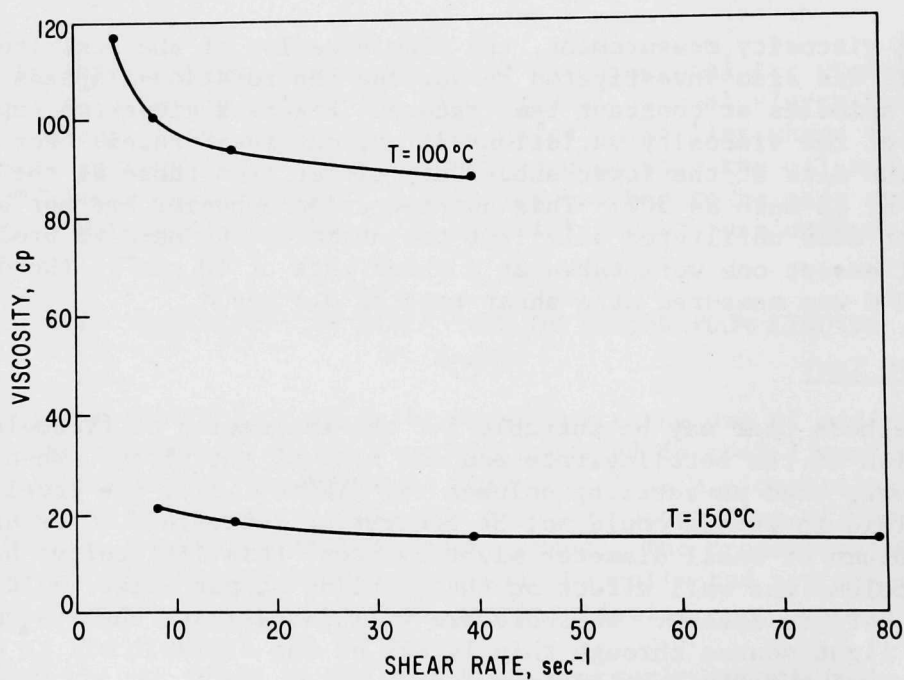


Fig. 2. Viscosity *vs.* Shear Rate for Centrifuged SYNTHOIL Oil



Two other methods may be adopted to evaluate flocculants for our viscous oil system. The first one,<sup>2</sup> widely used in England in sewage treatment, uses a capillary-suction apparatus which is commercially available. This apparatus consists of a small open-bottom cup (10- or 18-mm dia) placed on a standard filter paper (Whatman No. 17) which rests on a solid surface. Slurry is poured into the cup. A cake forms as the liquid flows through the slurry by capillary action. The cake resistance is measured by the suction time required for the filtrate to diffuse radially through the filter paper from an inner circle to an outer circle. Excellent correlation between the suction time and the settling rate in a graduated cylinder has been obtained.<sup>2</sup>

In the second method, the effect of the additives on the agglomerating tendencies of solids in an oil is examined with an oil immersion spectroscope. A slide is prepared by spreading a thin film of coal-derived oil onto a slide glass with a razor blade. A known amount of liquid additive is then dropped in the center of the oil film, and a cover glass is placed over the film. The material is then observed, using oil immersion techniques. Application of this method is limited to an additive soluble in the liquid phase. It is being used by researchers in Oak Ridge National Laboratory to study the effect of solvents on the agglomerating tendencies of solids in unfiltered oil from the COED process.

#### Particle-Size Analysis of SYNTHOIL Solids

Particle size analysis and characterization have long been recognized as efficient design tools in solid-liquid separation. We therefore plan to determine the size distribution of SYNTHOIL solids.

Solids extraction will be carried out in a Soxhlet extractor with various solvents or combinations of solvents--benzene, pentane, tetrahydrofuran, etc. The solids extracted will be examined, using either a Coulter Counter or an optical microscope. The ash content of the SYNTHOIL product will be determined by low-temperature ashing of the extracted solids.

#### IV. CATALYST TESTING (R. Lo)

The objective of this task is to evaluate new commercially available catalysts for use in the SYNTHOIL process. Testing will be carried out in a continuous unit at the operating conditions of SYNTHOIL. Results gained from this study will be used to identify the better catalysts. Subsequent screening of these selected catalysts will then be made on the long-life test unit at PERC to optimize catalyst selection for use in a 10 tons per day (TPD) SYNTHOIL process development unit currently under design.

This work will be performed in two phases.

##### A. Phase I. Design, Construction, and Shakedown of Test Unit

We have designed (in consultation with PERC) and will construct a continuous high-pressure coal liquefaction unit wherein catalysts may be tested under SYNTHOIL process conditions, 450°C and 2000-4000 psi. The unit will have the

capacity to process at least 4 oz/hr of coal slurry containing 35-50 percent coal in recycle oil. The design will include sufficient instrumentation and will be automated so that it can be operated continuously with minimal attention for periods of no less than 12 days.

#### (B) Phase II. Tests of Catalysts

We will select, with PERC's agreement, four or more commercially available hydrodesulfurization catalysts, and will test them on the catalyst test unit in 300-hr continuous runs to determine the intermediate life and product variability at 2000 psi and 4000 psi for each catalyst. Product samples will be collected at least once every 24 hr during the run and will be analyzed to determine: coal conversion; sulfur, nitrogen, and oxygen removal; hydrogen consumption; viscosity; and specific gravity of the product oil.

#### (C) Experimental System Design

We have held meetings with PERC personnel to discuss and review the design of the catalyst test equipment. A Program Evaluation and Review Technique (PERT) chart was also prepared, based on our estimation of the time requirement for each individual activity step and for the completion of this program within the time frame set forth by the work plan. In the PERT charge (Fig. 3), the status of this task on January 1, 1976, is indicated by arrows on the chart.

Design of the catalyst test unit has been completed. Components that have a long procurement lead time have been ordered. These components, some of which are similar to those described in Section II of this report, include a slurry circulation pump, two high-pressure slurry delivery pumps, a primary gas compressor, a recycle gas compressor, a preheater and a reactor furnace, a gas scrubber water delivery pump, two high-pressure gas-liquid separators, and two high-pressure surge/knockout tanks. The unit will be fabricated and assembled by a contractor. A safety and design review for this unit has been completed, and a purchase requisition has been submitted for bidding. We expect installation of the unit to be completed by the end of August, 1976.

The catalyst test unit is designed to process 0.5 to 5 lb slurry/hr. The unit will be located in an enclosure, with all monitoring and control done remotely via the control console, which will be located outside the enclosure. A general description of the catalyst test unit is presented in the following paragraphs. A schematic flow diagram of the unit is shown in Fig. 4.

The unit is divided into the following four functional subsystems:

1. Slurry delivery system
2. Gas delivery and recirculation system
3. Reactor system
4. Downstream separation and depressurization system

##### 1. Slurry Delivery System

The primary function of this system is to prepare and deliver a coal-oil slurry to the reactor section at a maximum system pressure of 4000 psig.

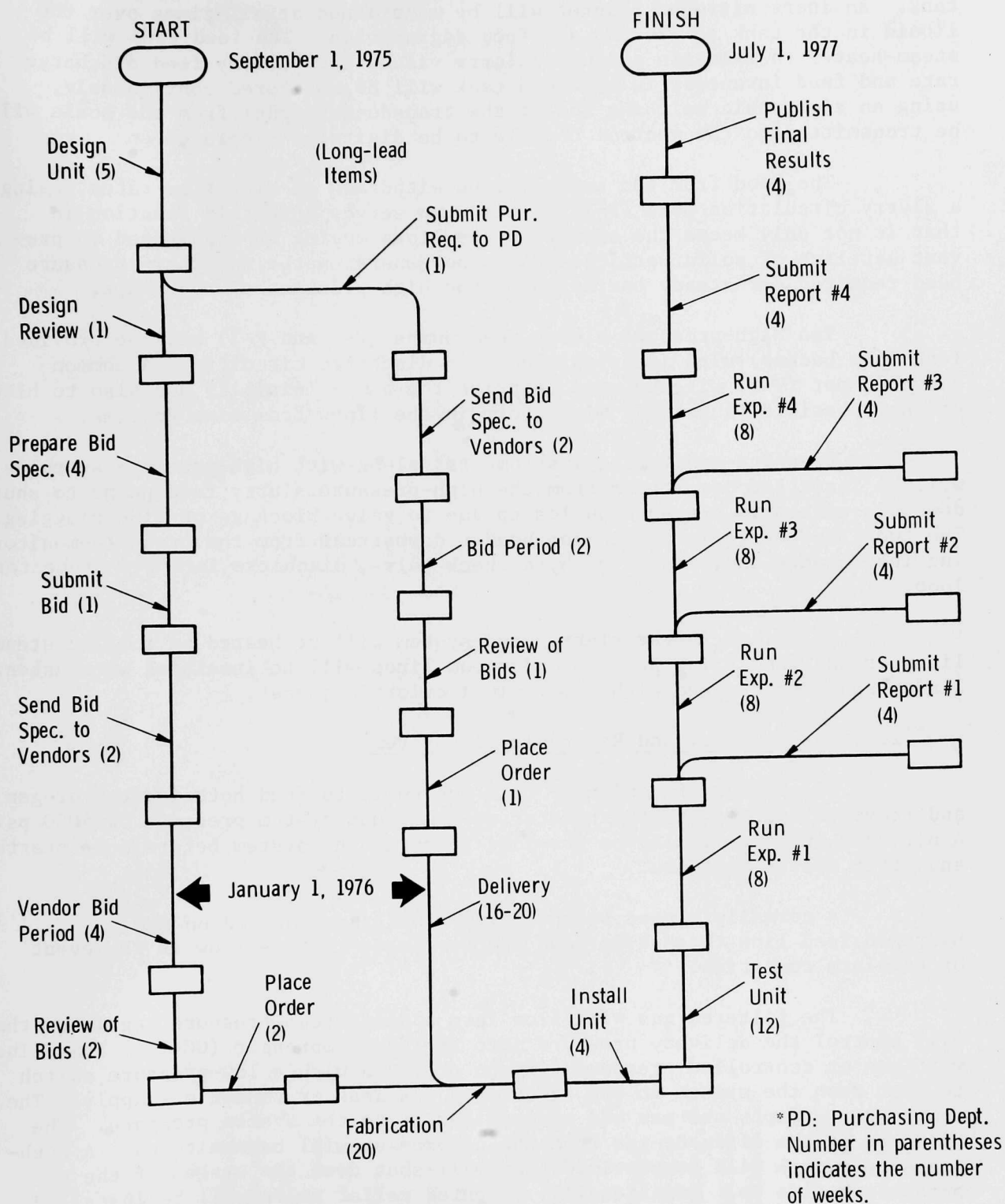


Fig. 3. PERT Chart for Catalyst Test Unit

A coal-oil mixture will be continuously stirred in a slurry feed tank. An inert nitrogen blanket will be maintained at all times over the liquid in the tank to prevent any feed degradation. The feed tank will be steam-heated to maintain adequate slurry viscosity. Slurry feed discharge rate and feed inventory in the feed tank will be monitored continuously, using an electronic weighing scale; the transducer signal from the scale will be transmitted to the control console to be digitally displayed.

The feed from the tank will be withdrawn at high flow rates, using a slurry circulation pump (P-1). This pump serves a twofold function in that it not only keeps the slurry in the lines moving at high speed to prevent settling of solid particles but also generates the necessary pressure head required for steady operation of the high-pressure slurry pumps.

Two high-pressure slurry feed pumps (P-2 and P-3) will be provided (one as a backup) with identical feed and discharge circuitry. A common blowdown pot (V-1) will be used to prime the pumps initially and also to blow off any possible plugs that might form in the lines from time to time.

Pressure-monitoring instruments, along with high-pressure switches, will be installed downstream from the high-pressure slurry feed pumps to shut down the system if pressure builds up due to valve blockage or line plugging. The two lines merge into a common header downstream from the pressure-monitoring instruments and, protected by a check valve, discharge into the preheater loop.

All lines in the slurry feed system will be heated by coiling steam lines around them. The process and steam lines will be insulated with asbestos lining and then covered with a polyvinyl chloride jacket.

## 2. Gas Delivery and Recirculation System

The primary function of this system is to feed both input hydrogen and recycled hydrogen to the reactor at a maximum system pressure of 4000 psig. A nitrogen input will also be provided to purge the system before unit startup and after system shutdown.

A normally closed solenoid valve will be provided on the hydrogen feed line to shut it down and terminate the gas flow in the event of an alarm condition.

The filtered gas will flow into a downstream pressure regulator that will control the delivery pressure into the gas compressor (GC-1). This line with gas at controlled pressure will be provided with a low-pressure switch to shut down the system in the event of a gas leak or runout of supply. The compressor accepts the gas and pressurizes it to the system pressure. The pressure of the effluent gas from the compressor will be monitored. A high-pressure switch will be provided that will shut down the system if the pressure builds up. Additionally, a vented relief valve will be installed for backup safety.

An electronic Delta-P transducer will be incorporated to measure incoming gas flow rates. A check valve will isolate this system from the rest of the process.



SYMBOL	SCHEDULE LISTING
	FORGED CONTROL VALVE
	ON-OFF BALL VALVE
	HIGH PRESSURE BONNET VALVE
	REGULATING/SHTOFF VALVE
	NEEDLE VALVE
	THROTTLING VALVE
	THREE WAY BALL VALVE
	CHECK VALVE
	SOLENOID VALVE
	RELIEF VALVE
	BACK PRESSURE REGULATOR
	PRESSURE GAUGE
	PRESSURE SWITCH
	HIGH PRESSURE ALARM
	LOW PRESSURE ALARM
	LIQUID LEVEL CONTROL
	PRESSURE TRANSDUCER
	DIAPHRAGM SEAL
	FILTER
	RUPTURE DISC
	DRY TEST METER
	GAS SNUBBER
	PRESSURE REGULATOR
	DELTA-P TRANSDUCER
P	PUMP
GC	GAS COMPRESSOR
V	PRESSURE VESSEL

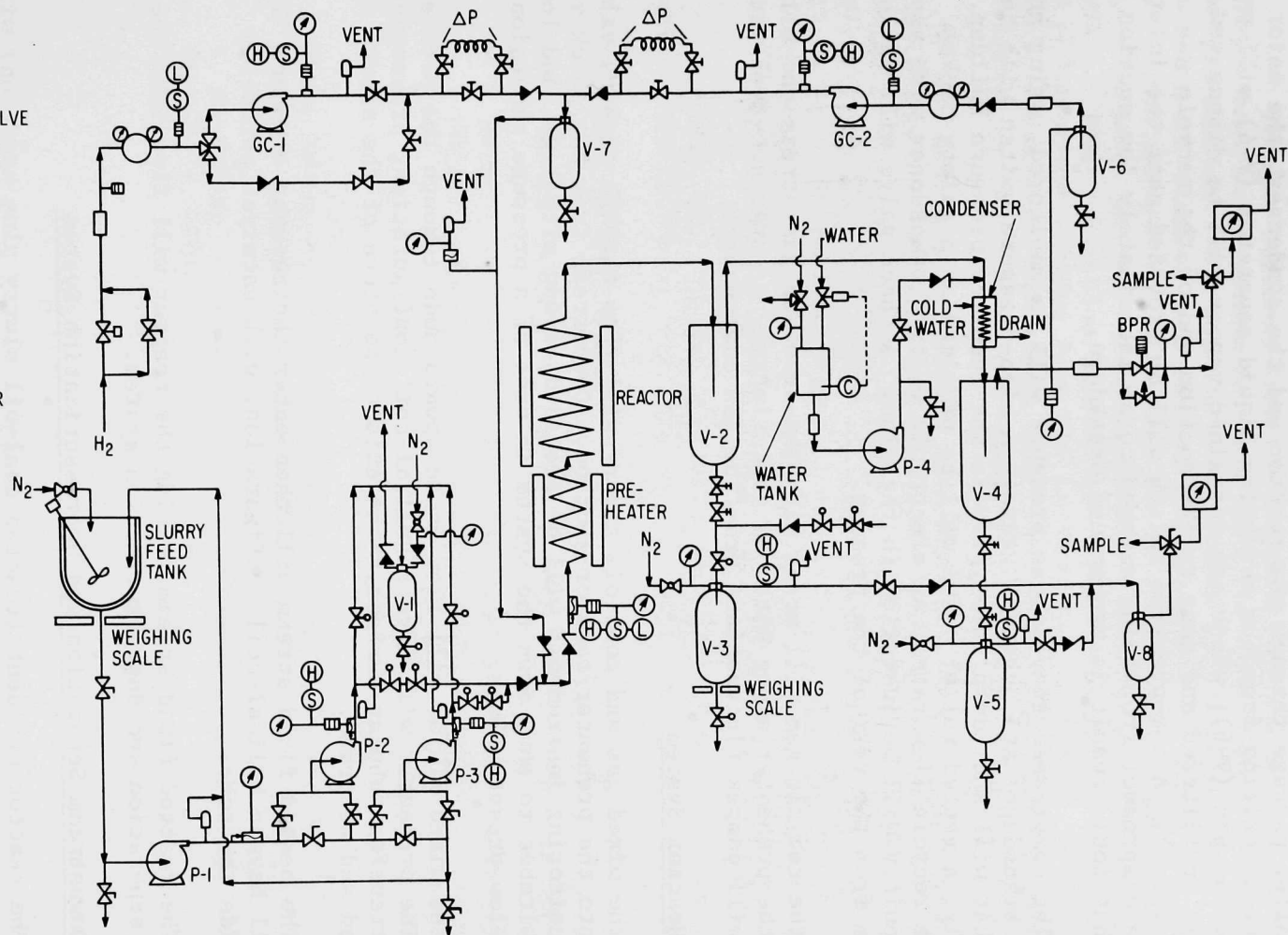


Fig. 4. Schematic Flow Diagram of the Catalyst Test Unit

After passage through the reactor and the condenser, the major hydrogen stream exiting from the scrubbing liquid separator (V-4) will pass into a knockout pot (V-6), where any entrained vapors will be disengaged. The gas will be filtered and sent to the suction end of the recycle gas compressor (GC-2). A low-pressure switch will be provided near the inlet to prevent the compressor from being exposed to an inordinately low suction pressure that could result in mechanical breakdown.

The compressed recycle gas pressure will be monitored, using an electronic transducer and pressure gauge. A high-pressure switch will be provided that will shut down the system in the event of pressure buildup. Additionally, a vented relief valve will be provided as a safety backup. To measure the recycle flow rates, an electronic Delta-P transducer with appropriate circuitry will be installed in the line. A check valve will isolate this system from the rest of the process.

The recycle gas will merge with the incoming gas stream and will flow into the preheater along with the coal slurry. A surge pot (V-7) in this line will dampen fluctuations from the gas compressors.

### 3. Reactor System

The mixed gas and coal-oil slurry will flow through a check valve and then into the preheater/reactor section. Downstream from the check valve, pressure-monitoring instruments will be provided, along with high- and low-pressure switches to shut down the system in case of a pressure excursion above or below preset values.

The mixed fluids will flow in an upward mode through the preheater furnace. The preheater will have a helical-coil configuration, presenting a long heat transfer tubular surface. The exit temperature of the slurry will be monitored and recorded.

The heated fluid stream will then enter the reactor element. The reactor will have a helical-coil configuration, with catalyst particles packed inside the tube.

The reacted fluid stream leaving the reactor will flow into the downstream separation and depressurization system.

### 4. Downstream Separation and Depressurization System

The reactor effluent (reacted coal-oil slurry plus hydrogen) will flow vertically down into the 5-gal high-pressure liquid separator (V-2). The liquid product will be drained to atmospheric pressure from the bottom of the receiver, using throttling drain valves. Two throttling valves will be provided because of the tremendous erosion of the valve seat and stem by the slurry during depressurization from 4000 psig to atmospheric pressure. The assumption is that it is unlikely that both valves will begin to leak simultaneously. The depressurized liquid from the separator will be collected in a 2-gal receiver (V-3), which for safety reasons has a medium pressure rating of 1800 psig.

The gas effluent from the separator (V-2) will be scrubbed with a high-pressure water stream injected into the line to remove ammonia and hydrogen sulfide. The gas and water stream will then be cooled through a condenser and flashed into another high-pressure separator (V-4). The water will be drained periodically from the bottom to atmospheric pressure and collected in a receiver (V-5) identical to the one which collects the oil product. Both collection vessels will be flushed with an inert gas stream. The effluent gas streams from the low-pressure receivers, protected by relief-shutoff-check valve circuitry, will flow into a common header where the stream can either be switched to the sample mode or through a gas meter and vented.

The hydrogen effluent from the scrubbing liquid separator (V-4) will be monitored for pressure and then split into two streams. A major fraction will go to the gas recycle section. A minor fraction, equivalent to the gas input through the feed compressor minus the hydrogen consumed in the reaction, will be filtered and discharged through the system back-pressure controller. The depressurized hydrogen gas stream, protected by a relief valve, can be switched either into a sample loop or sent through a gas meter and vented.

Both the catalyst test unit and the test unit for measuring heat transfer coefficients, Task (B), will be installed in the same facility.

#### D. Schedule of Deliverables

- a. Design, build, and install a Catalyst Test Unit by September, 1976.
- b. Deliver data on a total of four catalysts, one at a time at approximately two-month intervals from September 1976 to September, 1977.

#### REFERENCES

1. "Coal Technology Program Quarterly Progress Report No. 2," Oak Ridge National Laboratory (August 1975).
2. F. M. Tiller, J. Wilensky, and P. J. Farrell, Chem. Eng. 123-126 (April 29, 1974).





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